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(54) Title: **IMPROVED LOW-COST DENTIFRICE COMPOSITION**

(57) Abstract: An improved low-cost dentifrice composition comprises from about 6 % to about 70 % of an abrasive polishing material, from about 0.5 % to about 4.5 % of a thickener, from about 0.5 % to about 45 % of a hexahydric humectant, from about 0.05 % to about 2.5 % of an anti-cavity agent, and at least about 30 % of water. The composition comprises less than about 10 % dihydric and trihydric alcohols, and has a viscosity of from about 8 to about 100 BKU.

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IMPROVED LOW-COST DENTIFRICE COMPOSITION

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FIELD OF THE INVENTION

The present invention relates to an oral care composition. Specifically, the present invention relates to a low-cost dentifrice composition.

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BACKGROUND OF THE INVENTION

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Good oral hygiene may be at least in part achieved by brushing the teeth with an oral composition such as a dentifrice composition. A dentifrice composition is widely acknowledged as important in contributing to improving oral health, especially via a reduction in the incidence of dental cavities and the build-up of tartar and dental calculus. Such conditions result from oral plaque, and may lead to diseases such as periodontitis and gingivitis. These diseases remain a major cause of tooth loss in adults today. In addition, other oral health afflictions, such as staining of the tooth enamel and oral malodor (bad breath) may be reduced by regular tooth brushing with a dentifrice composition.

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Although a number of dentifrice compositions have been described and many types of dentifrices and other oral care compositions are now commercially available, there is still the desire and need to develop improved products for promoting oral health. In addition, current dentifrice compositions may require expensive ingredients, and or production techniques. Accordingly, such dentifrice compositions may be too expensive for certain locales, or for certain consumer segments. Accordingly, it is desirable to improve the cost-effectiveness of such products without decreasing their efficacy, so that they may become increasingly available to consumers of all income levels, and in all locations.

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Previous low cost dentifrice compositions typically contain an abrasive, a humectant, an aqueous solvent, at least about 15% dihydric and trihydric

alcohols, and a low level of water. However, these dentifrice compositions typically do not contain an anti-cavity agent, because they are typically expensive. Accordingly, such low-cost dentifrice compositions may not provide the desired level of anti-caries protection. In addition, high levels of dihydric and trihydric alcohols are typically present, because it is commercially important that a dentifrice composition possess acceptable physical properties, such as texture and rheology. Consumers typically require an acceptable dentifrice composition to be, for example, smooth, squeezable, and relatively non-sticky.

Accordingly, the need exists for a dentifrice composition that provides satisfactory oral health benefits in a cost-effective manner.

SUMMARY OF THE INVENTION

The present invention relates to an improved low-cost dentifrice composition comprising from about 6% to about 70% of an abrasive polishing material, from about 0.5% to about 4.5% of a thickener, from about 0.5% to about 45% of a hexahydric humectant, from about 0.05% to about 2.5% of an anti-cavity agent, and at least about 30% of water. The composition comprises less than about 10% dihydric and trihydric alcohols, and has a viscosity of from about 8 to about 100 BKU.

It has now been found that the present invention may provide an effective, stable, and less irritating composition which is both easy to produce and cost-effective. In addition, such an improved dentifrice composition provides oral health and/or cavity reduction which is comparable to, or improved over, current dentifrice compositions. Furthermore, the flavor release, rheology, and/or texture of the improved dentifrice composition may be comparable to, or improved over, current dentifrice compositions.

These and other features, aspects, advantages, and variations of the present invention, and the embodiments described herein, will become evident to those skilled in the art from a reading of the present disclosure with the appended claims, and are covered within the scope of these claims.

DETAILED DESCRIPTION OF THE INVENTION

All percentages, ratios and proportions herein are by weight of the final dentifrice composition, unless otherwise specified. All temperatures are in degrees Celsius (°C) unless otherwise specified. All documents cited are

incorporated herein by reference in their entireties. Citation of any reference is not an admission regarding any determination as to its availability as prior art to the claimed invention.

5 Herein, "alkyl" means a hydrocarbonyl moiety which is straight or branched, saturated or unsaturated. Unless otherwise specified, alkyl moieties are preferably saturated or unsaturated with double bonds, preferably with one or two double bonds. Included in the term "alkyl" is the alkyl portion of acyl groups.

10 Herein, "comprising" means that other steps and other components which do not affect the end result may be added. This term encompasses the terms "consisting of" and "consisting essentially of."

 Herein, "effective amount" means an amount of a compound or composition sufficient to significantly induce a positive benefit, preferably an oral health benefit, but low enough to avoid serious side effects, i.e., to provide a reasonable benefit to risk ratio, within the sound judgment of a skilled artisan.

15 Herein, "dentifrice composition" means a composition whose physical form is a paste or gel composition unless otherwise specified. The dentifrice composition may be in any desired aesthetic form, such as deep striped, surface striped, multi-layered, having the gel surrounding the paste, or any combination thereof. Alternatively, the dentifrice composition may be a dual-phase system
20 comprising two or more dentifrice compositions contained in a physically separated compartment of a dispenser and dispensed side-by-side.

 Herein, "dihydric alcohol" indicates a molecule which possess two hydroxy groups, and includes common humectants such as polyethylene glycol, propylene glycol, etc. Herein, "trihydric alcohol" indicates a molecule which
25 possess three hydroxy groups, and includes common humectants such as glycerine. Dihydric alcohols and trihydric alcohols are collectively referred to herein, as "dihydric and trihydric alcohols".

 Herein "dispenser" means any pump, tube, or container suitable for dispensing the dentifrice composition.

30 Herein, "substantially free" means that no such compound is purposely added as an ingredient to the dentifrice composition. However, this term does not preclude the possibility that a small amount of such a compound may be present as an impurity, or at a level which does not significantly affect the overall performance, cost, and/or stability of the dentifrice composition.

All ingredients such as actives and other ingredients useful herein may be categorized or described by their cosmetic and/or therapeutic benefit or their postulated mode of action. However, it is to be understood that the actives and other ingredients useful herein can, in some instance, provide more than one cosmetic and/or therapeutic benefit or operate via more than one mode of action. Therefore, classifications herein are made for the sake of convenience and are not intended to limit an ingredient to the particular stated application or applications listed.

Abrasive Polishing Material

The abrasive polishing material useful herein may be selected from any material which does not excessively abrade dentine. The abrasive polishing material preferably has a calcium content of less than about 23%. Typical abrasive polishing materials include silicas including gels and precipitates; aluminas; phosphates including orthophosphates, polymetaphosphates, and pyrophosphates; and mixtures thereof. Specific preferred examples of the abrasive polishing material useful herein include silicas (further described below), dicalcium orthophosphate dihydrate, calcium pyrophosphate, tricalcium phosphate, calcium polymetaphosphate, insoluble sodium polymetaphosphate, hydrated alumina, beta calcium pyrophosphate, calcium carbonate, resinous abrasive materials such as particulate condensation products of urea and formaldehyde, dicalcium phosphate, alumina trihydrate, insoluble sodium metaphosphate, and others such as disclosed by Cooley, et al., in U.S. Patent 3,070,510, issued Dec. 25, 1962. Mixtures of abrasives may also be used. While calcium carbonate may be used herein, it is not preferred as it may be incompatible with the anti-cavity agent, as described below; therefore, in a preferred embodiment, the dentifrice composition is substantially free of calcium carbonate.

Silica dental abrasives of various types are also highly preferred because of their unique benefits of exceptional dental cleaning and polishing performance without unduly abrading tooth enamel or dentine. The silica abrasive polishing materials herein, as well as other abrasive polishing materials, generally have an average particle size ranging between about 0.1 to about 30 microns, preferably from about 5 to about 15 microns, and have good fluoride compatibility (i.e. to provide soluble fluoride ions). The abrasive polishing material may be precipitated silica or silica gels such as the silica xerogels described in Pader, et

al., U.S. Patent 3,538,230, issued Mar. 2, 1970, and DiGiulio, U.S. Patent 3,862,307, issued Jan. 21, 1975. Preferred are the silica xerogels marketed under the trade name "Sylloid" by the W. R. Grace & Company, Davison Chemical Division. Also preferred are the precipitated silica materials such as those marketed by the J. M. Huber Corporation under the trade name, "Zeodent", particularly the silica carrying the designation "Zeodent 119". The types of silica dental abrasive polishing materials useful in the dentifrices of the present invention are described in more detail in Wason, U.S. Patent 4,340,583, issued July 29, 1982. The dentifrice composition comprises an abrasive polishing material at a level of from about 6% to about 70%, preferably from about 10% to about 50%, and more preferably from about 15% to about 30%, by weight of the dentifrice composition.

Thickener

The thickener useful herein acts as a binder to provide a desirable consistency. The thickeners useful in the present dentifrice may be selected from an inorganic gel-forming thickener, a modified cellulose thickener, a natural vegetable gum thickener, a bacterial gum thickener, a synthetic organic polymer thickener, a miscellaneous natural thickener, and a mixture thereof. Preferred inorganic gel-forming thickeners include a silica precipitate, a silica aerogel, a pyrogenic silica, a colloidal magnesium aluminum silicate, and a mixture thereof. Preferred modified cellulose thickeners include carboxymethyl cellulose, hydroxyethyl cellulose, hydroxymethyl carboxyethyl cellulose, hydroxypropyl cellulose, hydroxymethyl carboxypropyl cellulose, methyl cellulose, ethyl cellulose, and a mixture thereof. Such thickeners are available from, for example, Aqualon Co., Hopewell, VA, USA; Jiangmen Fenghua Fine Chemical Industry Co., Ltd., Jiangmen City, China; and Zhangjiagang Xinguang Chemical Factory, Zhangjiagang City, China. Preferred natural vegetable gum thickeners include Irish Moss (carrageenan), gum tragacanth, gum arabic, gum ghatti, gum acacia, locust bean gum, sodium alginate, and a mixture thereof. A preferred bacterial gum thickener is Xanthan gum. Preferred synthetic organic polymer thickeners include a polyacrylate, a polyvinyl pyrrolidone, and a mixture thereof, more preferably a cross-linked homopolymer of acrylic acid, such as carbomer. Highly preferred carbomers include those of the Carbopol series, especially Carbopol 956, available from BF Goodrich Co., Calvert City, Kentucky, USA.

Preferred miscellaneous natural thickeners include agar-agar, pectin, gelatin, and a mixture thereof. From a performance, economy, safety, and cost standpoint, an even more preferred thickener is a carboxymethyl cellulose, a carbomer, and a mixture thereof.

5 The thickener is present at a level of from about 0.5% to about 4.5%, preferably from about 0.5% to about 2.0%, and more preferably from about 0.8% to about 1.5%, by weight of the dentifrice composition.

Hexahydric Humectant

10 The present dentifrice composition contains a hexahydric humectant therein, which serves to keep toothpaste compositions from hardening upon exposure to air, and typically also impart a desirable sweetness of flavor to the dentifrice composition. The hexahydric humectant useful herein has six hydroxyl groups, and is typically, although not necessarily, a sugar, or derived from a
15 sugar. The hexahydric humectant may have a linear, branched, or cyclic backbone; and preferably the hexahydric humectant has a cyclic backbone of about 6 carbon atoms. The hexahydric humectant may also have one or more additional functional groups, such as carboxylic acid moieties, amine moieties, amido moieties, ester moieties, and mixtures thereof. More preferably, the
20 hexahydric humectant is selected from the group consisting of sorbitol, xylitol, glucose, and a mixture thereof, and even more preferably, sorbitol. Such sugars may further reduce the formulation cost, by reducing or eliminating the need for an optional sweetener.

 Without intending to be limited by theory, it is believed that these
25 hexahydric humectants, and especially sorbitol, xylitol, glucose, and mixtures thereof, interact with the thickeners described herein to provide superior water-binding abilities due to the accessibility and positioning of their multiple hydroxyl groups. It is further believed that such benefits are not present when, for example, the level of hexahydric humectant, alone, is increased. This synergistic
30 effect in turn may reduce the need for more expensive thickeners and dihydric and trihydric alcohols, and may further reduce formulation costs while maintaining an acceptable rheology and texture for the dentifrice composition. Such hexahydric humectants are also typically easily available, cost effective, safe in almost all instances, and are surprisingly stable when included in the present
35 dentifrice composition.

In addition, it is believed that the high hydrophilicity of such hexahydric humectants directly improves the flavor retention and flavor release of oil-based flavoring systems, and allows a lower level of flavoring to be used. As flavoring systems are typically highly distilled, and expensive mixtures, the present dentifrice composition may have significantly reduced formulation costs. Because oil-based flavoring systems are relatively insoluble in such a hydrophilic compound, more flavor is directly perceivable by a dentifrice composition user. In contrast, oil-based flavoring systems may be partially or completely soluble in dihydric and/or trihydric alcohols, such as glycerin and polyethylene glycol, which are common in dentifrice compositions; this in turn reduces the flavoring system's ability to be perceived by a dentifrice composition user, and may increase formulation costs by requiring that a greater level of flavoring system be included.

Without intending to be limited by theory, it is believed that the combination of the hexahydric humectant and the thickener described herein synergistically interact to provide acceptable rheology, aesthetics, stability, and equal or better performance, without the need for the traditionally high levels (e.g., 15%, or more) of dihydric and trihydric alcohols. As noted above, such dihydric and trihydric alcohols are typically expensive, and therefore the reduction, or removal of such compounds may significantly reduce the formulation costs of the dentifrice composition. Accordingly, the dentifrice composition of the present invention typically comprises less than about 10%, preferably between 0% and about 5% dihydric and trihydric alcohols. More preferably, the dentifrice composition of the present invention is substantially free of dihydric and trihydric alcohols.

The hexahydric humectant is typically present at a level of about 0.5% to about 45%, preferably from about 5% to about 40%, and more preferably from about 10% to about 35%, by weight of the dentifrice composition.

Anti-Cavity Agent

The decrease in formulation costs due to the reduction or elimination of dihydric and trihydric alcohols allows a formulator greater freedom to include, and/or increase the level of an anti-cavity agent in the dentifrice composition, without having to raise the selling price. In certain countries and locales, this may increase the availability of good, effective oral health care to a significant fraction

of the general populace. The anti-cavity agent typically contains a fluoride ion, preferably a water-soluble fluoride ion. Fluoride ion sources are well known for use in oral care compositions as anti-carries agents. Fluoride ions are contained in a number of oral care compositions for this purpose, particularly in dentifrice compositions such as toothpastes. Patents disclosing such dentifrice compositions include U.S. Pat. No. 3,538,230, Nov. 3, 1970 to Pader, et al.; U.S. Pat. No. 3,689,637, Sept. 5, 1972 to Pader; U.S. Pat. No. 3,711,604, Jan 16, 1973 to Colodney, et al.; U.S. Pat. No. 3,911,104, Oct. 7, 1975 to Harrison; U.S. Pat. No. 3,935,306, Jan. 27, 1976 to Roberts, et al.; and U.S. Pat. No. 4,040,858, Aug. 9, 1977 to Wason.

Application of fluoride ions to dental enamel serves to protect teeth against decay. A wide variety of fluoride ion-yielding materials may be employed as sources of soluble fluoride in the instant compositions. Examples of suitable fluoride ion-yielding materials are found in Briner, et al.; U.S. Pat. No. 3,535,421; issued Oct. 20, 1970 and Widder, et al.; U.S. Pat. No. 3,678,154; issued July 18, 1972. Preferred fluoride ion sources for use herein include sodium fluoride, potassium fluoride, ammonium fluoride, sodium monofluorophosphate, stannous fluoride, and a mixture thereof. Sodium fluoride is particularly preferred, and is available from, for example, Jinan Chemical Sub-factory, Jinan City, Shandong province, China. Preferably the dentifrice composition provides from about 50 ppm to 10,000 ppm, more preferably from about 100 to 3000 ppm, of fluoride ions in the compositions that contact dental surfaces when used with the delivery system of the present invention. Such fluoride ion measurements are typically determined from a 3:1 slurry of water and dentifrice composition. Generally, the anti-cavity agent, preferably the fluoride ion anti-cavity agent, will be present at a level of from about 0.05% to about 2.5%, preferably from about 0.1% to about 1%, and more preferably from about 0.1% to about 0.5%, by weight of the composition.

However, when using fluoride ions as the anti-cavity agent, care should be taken to reduce interactions with incompatible materials such as calcium carbonate, which may be present as an abrasive polishing material. Accordingly, if a fluoride ion is present, and especially if sodium fluoride is present, then it is highly preferred that the dentifrice composition be substantially free of calcium carbonate. While fluoride ion-containing anti-cavity agents are preferred, non-fluoride anti-cavity agents known in the art, such as monosodium phosphate,

monohydrate, are also useful herein, either alone, or in combination with a fluoride ion-containing anti-cavity agent.

Water

5 The dentifrice composition further contains at least 30% by weight of the dentifrice composition. As described and measured herein, "water" includes water from solutions (i.e., a humectant solution, a sweetener solution, a surfactant solution, etc.), but not bound water (i.e., from hydrates at room temperature). Prior to addition to the dentifrice composition, the water useful
10 herein is typically purified, or distilled to remove impurities.

Adjunct Compound

 The dentifrice composition may further contain one or more adjunct compounds known in the art, preferably an adjunct compound selected from the
15 group consisting of a dihydric or trihydric humectant, a buffering agent, a peroxide source, an alkali metal bicarbonate source, an anti-tartar agent, a surfactant, titanium oxide, an antioxidant, a coloring agent, a flavoring system, a sweetening agent, an herbal agent, an anti-microbial agent, and a mixture thereof.

20 The present invention may comprise up to 10% of a dihydric or trihydric humectant, such as are traditionally used in dentifrice compositions. However, in a preferred embodiment, the dentifrice composition comprises between 0% and about 5% dihydric and trihydric alcohols. More preferably, the dentifrice composition of the present invention is substantially free of dihydric and trihydric
25 alcohols. Suitable humectants for use in the invention include glycerin, polyethylene glycol, propylene glycol, other edible polyhydric alcohols, and a mixture thereof, and preferably glycerin, polyethylene glycol, and a mixture thereof.

 The present composition typically contains a buffering agent. If the
30 dentifrice composition is a dual phase system, a buffering agent will be present in both dentifrice compositions. Buffering agents, as used herein, refer to agents that may be used to adjust the pH of the compositions to a range of about pH 4 to about pH 10, with pH of about 4 to about 8 being preferred, and pH of about 5.5 to about 8 being more preferred. These agents include alkali metal hydroxides,
35 carbonates, sesquicarbonates, borates, silicates, phosphates, imidazole, and

mixtures thereof. Specific buffering agents include monosodium phosphate, trisodium phosphate, sodium hydroxide, potassium hydroxide, alkali metal carbonate salts, sodium carbonate, imidazole, pyrophosphate salts, citric acid, and sodium citrate. Buffering agents are used at a level of from about 0.01% to about 30%, preferably from about 0.1% to about 10%, and more preferably from about 0.3% to about 7%, by weight of the present composition. Preferred buffering agents are monosodium phosphate, trisodium phosphate, pyrophosphate salts, citric acid, sodium citrate, and mixtures thereof. It has surprisingly been found that the dentifrice composition of the present invention does not require as high a level of buffering salts, as traditional dentifrice compositions. Without intending to be limited by theory, it is believed that this may result in reduced irritation to gums, and the oral cavity of the user.

Pyrophosphate salts may be present as buffering agents. The pyrophosphate salts useful in the present compositions include the dialkali metal pyrophosphate salts, tetra alkali metal pyrophosphate salts, and mixtures thereof. Sodium acid pyrophosphate, disodium dihydrogen pyrophosphate ($\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$), tetrasodium pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7$), and tetrapotassium pyrophosphate ($\text{K}_4\text{P}_2\text{O}_7$) in their unhydrated as well as hydrated forms are the preferred species. In compositions of the present invention, the pyrophosphate salt may be present in one of three ways: predominately dissolved, predominately undissolved, or a mixture of dissolved and undissolved pyrophosphate.

Compositions comprising predominately dissolved pyrophosphate refer to compositions where at least one pyrophosphate ion source is present. Free pyrophosphate ions may be present in a variety of protonated states depending on the pH of the composition.

Compositions comprising predominately undissolved pyrophosphate refer to compositions containing no more than about 20% of the total pyrophosphate salt dissolved in the composition, preferably less than about 10% of the total pyrophosphate dissolved in the composition. Tetrasodium pyrophosphate salt and tetrapotassium pyrophosphate salt are the preferred pyrophosphate salts in these compositions, with tetrasodium pyrophosphate salt being the more preferred. Tetrasodium pyrophosphate may be the anhydrous salt form or the decahydrate form, or any other species stable in solid form in the dentifrice compositions. The salt is in its solid particle form, which may be its crystalline and/or amorphous state, with the particle size of the salt preferably being small

enough to be aesthetically acceptable and readily soluble during use. The amount of pyrophosphate salt useful in making these compositions is any tartar control effective amount. Some or all of the tetrasodium pyrophosphate may be undissolved in the product and present as tetrasodium pyrophosphate particles.

5 Pyrophosphate ions in different protonated states (e.g., $\text{HP}_2\text{O}_7^{-3}$) may also exist depending upon the pH of the composition and if part of the tetrasodium pyrophosphate is dissolved.

Compositions may also comprise a mixture of dissolved and undissolved pyrophosphate salts. Any of the above mentioned pyrophosphate salts may be

10 used. The pyrophosphate salts are described in more detail in Kirk & Othmer, Encyclopedia of Chemical Technology, Third Edition, Volume 17, Wiley-Interscience Publishers (1982).

The present invention may include a peroxide source for whitening effect. The peroxide source is selected from the group consisting of hydrogen peroxide, calcium peroxide, urea peroxide, and mixtures thereof. The preferred peroxide

15 source is urea peroxide. The following amounts represent the amount of peroxide raw material, although the peroxide source may contain ingredients other than the peroxide raw material. The present composition may contain from about 0.01% to about 10%, preferably from about 0.1% to about 5%, more

20 preferably from about 0.2% to about 3%, and even more preferably from about 0.3% to about 0.8% of a peroxide source, by weight of the composition.

The present invention may also include an alkali metal bicarbonate salt. Alkali metal bicarbonate salts are soluble in water and unless stabilized, tend to release carbon dioxide in an aqueous system. Sodium bicarbonate, also known

25 as baking soda, is the preferred alkali metal bicarbonate salt. The alkali metal bicarbonate salt also may function as a buffering agent. The present composition may contain from about 0.5% to about 50%, preferably from about 0.5% to about 30%, more preferably from about 2% to about 20%, and most preferably from about 5% to about 18% of an alkali metal bicarbonate salt, by weight of the oral

30 composition.

Anti-tartar agents known for use in dental care products include phosphates. Phosphates include pyrophosphates, polyphosphates, polyphosphonates and mixtures thereof. Pyrophosphates are among the best known for use in dental care products. Pyrophosphate and polyphosphate ions

35 are delivered to the teeth derive from pyrophosphate polyphosphate salts. The

pyrophosphate salts useful in the present compositions include the dialkali metal pyrophosphate salts, tetra-alkali metal pyrophosphate salts, and mixtures thereof. Sodium acid pyrophosphate, disodium dihydrogen pyrophosphate ($\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$), tetrasodium pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7$), and tetrapotassium pyrophosphate ($\text{K}_4\text{P}_2\text{O}_7$) in their unhydrated as well as hydrated forms are the preferred species. While any of the above mentioned pyrophosphate salts may be used, tetrasodium pyrophosphate salt is preferred. Sodium polyphosphate and triethanolamine polyphosphates, for example, are preferred.

The pyrophosphate salts are described in more detail in Kirk & Othmer, Encyclopedia of Chemical Technology, Third Edition, Volume 17, Wiley-Interscience Publishers (1982). Additional anti-calculus agents include pyrophosphates or polyphosphates disclosed in U.S. Patent No. 4,590,066 issued to Parran & Sakkab on May 20, 1986; polyacrylates and other polycarboxylates such as those disclosed in U.S. Patent No. 3,429,963 issued to Shedlovsky on February 25, 1969 and U.S. Patent No. 4,304,766 issued to Chang on December 8, 1981; and U.S. Patent No. 4,661,341 issued to Benedict & Sunberg on April 28, 1987; polyepoxysuccinates such as those disclosed in U.S. Patent No. 4,846,650 issued to Benedict, Bush & Sunberg on July 11, 1989; ethylenediaminetetraacetic acid as disclosed in British Patent No. 490,384 dated February 15, 1937; nitrilotriacetic acid and related compounds as disclosed in U.S. Patent No. 3,678,154 issued to Widder & Briner on July 18, 1972; polyphosphonates as disclosed in U.S. Patent No. 3,737,533 issued to Francis on June 5, 1973, U.S. Patent No. 3,988,443 issued to Ploger, Schmidt-Dunker & Gloxhuber on October 26, 1976 and U.S. Patent No. 4,877,603 issued to Degenhardt & Kozikowski on October 31, 1989. Anticalculus phosphates include potassium and sodium pyrophosphates; sodium tripolyphosphate; diphosphonates, such as ethane-1-hydroxy-1,1-diphosphonate, 1-azacycloheptane-1,1-diphosphonate, and linear alkyl diphosphonates; linear carboxylic acids; and sodium zinc citrate.

Anti-tartar agents that may be used in place of, or in combination with the pyrophosphate salt include such known materials as synthetic anionic polymers including polyacrylates and copolymers of maleic anhydride or acid and methyl vinyl ether (e.g., Gantrez), as described, for example, in U.S. Patent 4,627,977, to Gaffar, et al.; as well as, e.g., polyamino propane sulfonic acid (AMPS), zinc citrate trihydrate, polyphosphates (e.g., tripolyphosphate; hexametaphosphate),

diphosphonates (e.g., EHDP; AHP), polypeptides (such as polyaspartic and polyglutamic acids), and mixtures thereof.

The present compositions may also comprise a surfactant, also commonly referred to as a sudsing agent. Suitable surfactants are those which are reasonably stable and foam throughout a wide pH range. The surfactant may be anionic, nonionic, amphoteric, zwitterionic, cationic, or mixtures thereof. Anionic surfactants useful herein include the water-soluble salts of alkyl sulfates having from 8 to 20 carbon atoms in the alkyl radical (e.g., sodium alkyl sulfate) and the water-soluble salts of sulfonated monoglycerides of fatty acids having from 8 to 20 carbon atoms. Sodium lauryl sulfate and sodium coconut monoglyceride sulfonates are examples of anionic surfactants of this type. Sodium Lauryl sulfate is available from, for example, Rhodia Specialty Chemical Wuxi company, Wuxi City, Jiangsu province, China. Other suitable anionic surfactants are sarcosinates, such as sodium lauroyl sarcosinate, taurates, sodium lauryl sulfoacetate, sodium lauroyl isethionate, sodium laureth carboxylate, and sodium dodecyl benzenesulfonate. Mixtures of anionic surfactants may also be employed. Many suitable anionic surfactants are disclosed by Agricola, et al., U.S. Patent 3,959,458, issued May 25, 1976. Nonionic surfactants which may be used in the compositions of the present invention may be broadly defined as compounds produced by the condensation of alkylene oxide groups (hydrophilic in nature) with an organic hydrophobic compound which may be aliphatic or alkyl-aromatic in nature. Examples of suitable nonionic surfactants include poloxamers (sold under trade name Pluronic), polyoxyethylene, polyoxyethylene sorbitan esters (sold under trade name Tweens), fatty alcohol ethoxylates, polyethylene oxide condensates of alkyl phenols, products derived from the condensation of ethylene oxide with the reaction product of propylene oxide and ethylene diamine, ethylene oxide condensates of aliphatic alcohols, long chain tertiary amine oxides, long chain tertiary phosphine oxides, long chain dialkyl sulfoxides, and mixtures of such materials. The amphoteric surfactants useful in the present invention may be broadly described as derivatives of aliphatic secondary and tertiary amines in which the aliphatic radical may be a straight chain or branched and wherein one of the aliphatic substituents contains from about 8 to about 18 carbon atoms and one contains an anionic water-solubilizing group, e.g., carboxylate, sulfonate, sulfate, phosphate, or phosphonate. Other suitable amphoteric surfactants are betaines, specifically cocamidopropyl betaine.

Mixtures of amphoteric surfactants may also be employed. Many of these suitable nonionic and amphoteric surfactants are disclosed by Gieske, et al., U.S. Patent 4,051,234, issued September 27, 1977. The present composition typically comprises one or more surfactants each at a level of from about 0.25% to about 12%, preferably from about 1% to about 10%, and more preferably from about 1% to about 8%, by weight of the composition.

Titanium dioxide may also be added to the dentifrice composition. Titanium dioxide is a white powder which adds opacity to the compositions. Titanium dioxide generally comprises from about 0.1% to about 5%, by weight of the composition.

An antioxidant is generally recognized as useful in the present composition, and may be included herein. Antioxidants are disclosed in texts such as Cadenas and Packer, The Handbook of Antioxidants, © 1996 by Marcel Dekker, Inc. Antioxidants that may be included in the oral care composition or substance of the present invention include, but are not limited to Vitamin E, ascorbic acid (vitamin C), uric acid, carotenoids, Vitamin A, flavonoids, herbal antioxidants, melatonin, aminoindoles, lipoic acids and mixtures thereof. Vitamins E and C are preferred, with levels of from about 0.01% to about 0.10% being desirable, and about 0.01% to about 0.05% being preferred.

One or more coloring agents may also be added to the present composition. The coloring agent may be in the form of an aqueous solution, preferably 1% coloring agent in a solution of water. Color solutions generally comprise from about 0.01% to about 5%, by weight of the composition.

A flavoring system may also be added to the compositions. Suitable flavoring components include tea mint, oil of wintergreen, oil of peppermint, oil of spearmint, clove bud oil, menthol, anethole, methyl salicylate, eucalyptol, cassia, 1-menthyl acetate, sage, eugenol, parsley oil, oxanone, alpha-irisone, marjoram, lemon, orange, propenyl guaethol, cinnamon, vanillin, ethyl vanillin, heliotropine, 4-cis-heptenal, diacetyl, methyl-para-tert-butyl phenyl acetate, and mixtures thereof. Non-limiting examples of commercially available flavoring systems include those sold under the trade names "Smoothmint," "Ultimint," and "Icy Mint," available from International Flavors and Fragrances, USA & China. Coolants may also be part of the flavoring system. Preferred coolants in the present compositions are the paramenthan carboxamide agents such as N-ethyl-p-menthan-3-carboxamide (known commercially as "WS-3") and mixtures thereof.

A flavoring system is generally used in the compositions at levels of from about 0.001% to about 5%, by weight of the composition.

A sweetening agent may be added to the present composition, such as saccharin, dextrose, sucrose, lactose, maltose, levulose, aspartame, sodium cyclamate, D-tryptophan, dihydrochalcones, acesulfame, and mixtures thereof. Sweetening agents are generally used in toothpastes at levels of from about 0.005% to about 5%, by weight of the composition.

An herbal agent, including but not limited to, golden thread extract, honeysuckle extract, and a mixture thereof, may also be present in the compositions herein at levels of from about 0.01% to about 0.05%. Such herbal agents are believed to provide anti-bacterial efficacy.

While not required, traditional anti-microbial agents may also be present in the dentifrice composition of the present invention. However, as they tend to be expensive, and as the present invention shows surprising stability without them, a preferred embodiment of the present invention is substantially free of traditional anti-microbial agents. A preferred traditional anti-microbial agent is 5-chloro-2-(2,4-dichlorophenoxy)-phenol, commonly referred to as triclosan, and is described in The Merck Index, 11th ed. (1989), p. 1529 (entry no. 9573), in U.S. Patent No. 3,506,720, and in European Patent Application No. 0,251,591 of Beecham Group, PLC, published January 7, 1988. Other specific antimicrobial agents include chlorhexidine, triclosan monophosphate, and essential oils including thymol, geraniol, carvacrol, citral, hinokitiol, eucalyptol, and mixtures thereof. Triclosan and other agents of this type are disclosed in Parran, Jr., et al., U.S. Patent 5,015,466, issued May 14, 1991, and U.S. Patent 4,894,220, Jan. 16, 1990 to Nabi, et al. These agents may be present at levels of from about 0.01% to about 1.5%, by weight of the composition.

Also included among such other anti-microbial agents are water insoluble non-cationic antimicrobial agents such as halogenated diphenyl ethers, phenolic compounds including phenol and its homologs, mono and poly-alkyl and aromatic halophenols, resorcinol and its derivatives, bisphenolic compounds and halogenated salicylanilides, benzoic esters, and halogenated carbanilides. The water soluble anti-microbials include quaternary ammonium salts and bis-biquanide salts, among others. Triclosan monophosphate is also a suitable water soluble antimicrobial agent. The quaternary ammonium agents include those in which one or two of the substitutes on the quaternary nitrogen has a carbon chain

length (typically alkyl group) from about 8 to about 20, typically from about 10 to about 18 carbon atoms while the remaining substitutes (typically alkyl or benzyl group) have a lower number of carbon atoms, such as from about 1 to about 7 carbon atoms, typically methyl or ethyl groups. Dodecyl trimethyl ammonium bromide, tetradecylpyridinium chloride, domiphen bromide, N-tetradecyl-4-ethyl pyridinium chloride, dodecyl dimethyl (2-phenoxyethyl) ammonium bromide, benzyl dimethylstearyl ammonium chloride, cetyl pyridinium chloride, quaternized 5-amino-1,3-bis(2-ethyl-hexyl)-5-methyl hexa hydropyrimidine, benzalkonium chloride, benzethonium chloride and methyl benzethonium chloride are exemplary of typical quaternary ammonium antibacterial agents. Other compounds are bis[4-(R-amino)-1-pyridinium] alkanes as disclosed in U.S. Patent 4,206,215, issued June 3, 1980, to Bailey. Stannous salts such as stannous pyrophosphate and stannous gluconate and other anti-microbials such as copper bisglycinate, copper glycinate, zinc citrate, and zinc lactate may also be included. Also useful are enzymes, including endoglycosidase, papain, dextranase, mutanase, and mixtures thereof. Such agents are disclosed in U.S. Patent 2,946,725, Jul. 26, 1960, to Norris, et al., and in U.S. Patent 4,051,234, September. 27, 1977 to Gieske, et al., incorporated herein by reference.

Also suitable for use as anti-microbial agents herein are phthalic acid and its salts including, but not limited to those disclosed in U.S. Pat. 4,994,262, Feb. 19, 1991, preferably magnesium monopotassium phthalate, chlorhexidine (Merck Index, no. 2090), alexidine (Merck Index, no. 222; hexetidine (Merck Index, no. 4624); sanguinarine (Merck Index, no. 8320); benzalkonium chloride (Merck Index, no. 1066); salicylanilide (Merck Index, no. 8299); domiphen bromide (Merck Index, no. 3411); cetylpyridinium chloride (CPC) (Merck Index, no. 2024; tetradecylpyridinium chloride (TPC); N-tetradecyl-4-ethylpyridinium chloride (TDEPC); octenidine; delmopinol, octapinol, and other piperidino derivatives; nicin preparations; zinc/stannous ion agents; antibiotics such as augmentin, amoxicillin, tetracycline, doxycycline, minocycline, and metronidazole; and analogs and salts of the above; methyl salicylate; hydrogen peroxide; metal salts of chlorite and mixtures thereof.

Method of Use

In practicing the present invention, the user need only apply the dentifrice composition herein, to the tooth surfaces of a human or animal, in the areas desired, in order to obtain a desired effect, e.g., whitening, breath freshening, caries prevention, pain relief, gum health, tartar control, etc. The compositions may also be applied to other oral cavity surfaces, such as the gingival or mucosal tissues, although it is believed that the benefits are best achieved when the dentifrice compositions are applied to the teeth. The dentifrice composition may contact the tooth and/or oral cavity surface either directly, or indirectly; however, it is preferred that the dentifrice composition be directly applied. The dentifrice composition may be applied by any means, but is preferably applied with a brush.

Method of Manufacture

The dentifrice composition may be formed by traditional means and techniques known in the art. For example, the hexahydric humectant, and any optional dihydric and trihydric alcohols, may be added to purified water, and homogenized with the anti-cavity agent in a blender, for about 2 minutes. A sweetening agent, an anti-microbial agent, and other adjunct compounds (other than the flavoring system, surfactant, and titanium oxide) may then be added, and the mixture homogenized for an additional 10 minutes. The thickener and titanium oxide are then added into the mixture, under vacuum of about 0.1-0.2 bar, for about 20 minutes to remove air bubbles. Surfactant, and flavoring are then added, and homogenized under vacuum for from 15 to 30 minutes to form the final dentifrice composition. The dentifrice composition is then packaged in individual dispensers, e.g., toothpaste tubes, for shipment and sale.

The final dentifrice composition typically has a viscosity of from about 8 to about 100 BKU, preferably from about 15 to about 80 BKU, and more preferably from about 15 to about 50 BKU. As used herein, "BKU" is the unit of viscosity. The viscometer is Brookfield viscometer, Model 1/2 RVT (1/2 spring strength), with a Brookfield "Helipath" stand. The spindle is a conventional "E-series" T-shaped spindle. The viscometer is placed on the Helipath stand and leveled via spirit levels. The E spindle is attached, and the viscometer is set to 2.5 RPM while it is running. The viscosity is measured after 10 minutes and the temperature is constant, at 25 °C. Commercially acceptable dentifrice compositions typically have an acceptable rheology, good texture, pleasing aesthetic characteristics, and a specific gravity greater than water, preferably

from about 1 to 1.5 g/mL, more preferably from about 1.1 to about 1.3 g/mL. A dentifrice having the above viscosity and specific gravity are usually acceptable to consumers.

5 Examples of the invention are set forth hereinafter by way of illustration and are not intended to be in any way limiting of the invention.

EXAMPLES 1-2

The following dentifrice compositions are prepared according to the method described above.

Component	Comparative Example A	Example 1	Example 2
Sodium fluoride, USP	0.25	0.25	0.25
Silica, precipitated (Zeodent 119)	20	20	20
Sorbitol	30.4	21	14
Purified water *	29.4	53.85	60.8
Triclosan, USP	0.05	--	0.05
Carbopol 956	0.2	0.3	0.3
Sodium carboxymethyl cellulose	.85	1	1
Sodium saccharin, USP	0.15	0.25	0.25
Trisodium phosphate, dodecahydrate	1.3	0.5	0.5
Sodium alkyl sulfate	2.1	1.8	1.8
Flavoring system	0.8	0.8	0.8
Titanium dioxide, USP (Rutile)	0.5	0.25	0.25
Glycerin	10	--	--
Polyethylene glycol	4	--	--
TOTAL	100	100	100
Brookfield viscosity (BKU) (after 10 minutes)	13-37	18	15
pH	6.8-7.4	6.8	6.8
Soluble fluoride level (3:1 slurry of purified water:dentifrice composition)	265-315 ppm	268 ppm	268 ppm
Specific gravity (g/mL)	1.32-1.38	1.2	1.2

* includes water from sorbitol solution, surfactant solution, etc.

- 5 Nonlimiting examples of the dentifrice compositions of the invention are described by Examples 1 and 2, and a Comparative Example A is also provided. All three formulations provide surprisingly good rheology and stability; however, the per-unit formulation cost of the Comparative Example A is about 50% greater than the formulation costs of Examples 1 and 2. Furthermore, Examples 1 and 2

- possess excellent stability even though they contain more than 50% water. Furthermore, Examples 1 and B provide improved flavor release (i.e., perceptibility of flavor), at the same flavoring system level as Comparative Example A. Surprisingly, after 1 month, the microbial growth of Example 1 is comparable to that of the Comparative Example A, and Example 2, even though Example 1 lacks Triclosan.

EXAMPLES 3-4

- The following dentifrice compositions are prepared according to the method described above.

Component	Example 3	Example 4
Sodium fluoride, USP	0.243	0.243
Silica, precipitated (Zeodent 119)	22	21
Sorbitol	30	30
Purified water *	32.657	38.057
Carbopol 956	0.3	0.3
Sodium carboxymethyl cellulose	1	1.1
Sodium saccharin, USP	0.25	0.25
Trisodium phosphate, dodecahydrate	3	0.5
Monosodium phosphate	2	—
Sodium alkyl sulfate	7.5	7.5
Flavoring system	0.8	0.8
Titanium dioxide, USP (Rutile)	0.25	0.25
TOTAL	100	100
Brookfield viscosity (BKU) (after 10 minutes)	21	18
pH	6.8	6.8
Soluble fluoride level (3:1 slurry of purified water:dentifrice composition)	268 ppm	268 ppm
Specific gravity (g/mL)	1.29	1.22

* includes water from sorbitol solution, surfactant solution, etc.

WHAT IS CLAIMED IS:

1. A dentifrice composition comprising:
 - A. from about 6% to about 70% of an abrasive polishing material;
 - B. from about 0.5% to about 4.5% of a thickener;
 - C. from about 0.5% to about 45% of a hexahydric humectant;
 - D. from about 0.05% to about 2.5% of an anti-cavity agent; and
 - E. at least about 30% of water,wherein the composition comprises less than about 10% dihydric and trihydric alcohols, and wherein the viscosity of the composition is from about 8 to about 100 BKU.
2. The dentifrice composition of Claim 1, wherein the thickener is selected from the group consisting of carboxymethyl cellulose, carbomer, Xanthan gum, and mixtures thereof.
3. The dentifrice composition of Claim 1, wherein the composition comprises at least 40% water.
4. The dentifrice composition of Claim 1, wherein the composition comprises from 0% to 5% dihydric and trihydric alcohols.
5. The dentifrice composition of Claim 1, wherein the hexahydric humectant is selected from the group of sorbitol, xylitol, glucose, and mixtures thereof.
6. The dentifrice composition of Claim 1, wherein the composition is substantially free of dihydric and trihydric alcohols.
7. The dentifrice composition of Claim 1, wherein the viscosity of the composition is from about 15 to about 80 BKU.
8. The dentifrice composition of Claim 1, further comprising an adjunct compound selected from the group consisting of a peroxide source, an alkali metal bicarbonate source, an anti-tartar agent, a surfactant, titanium dioxide, an antioxidant, a coloring agent, a flavoring system, a sweetening agent, an herbal agent, an anti-microbial agent, and a mixture thereof.

9. A method for cleaning a dental surface of a human or an animal, comprising the step of contacting the dental surface with the dentifrice composition according to Claim 1.
10. A dentifrice composition comprising:
 - A. from about 6% to about 70% of a silica abrasive polishing material;
 - B. from about 0.5% to about 4.5% of a thickener selected from the group consisting of carboxymethyl cellulose, carbomer, Xanthan gum, and mixtures thereof;
 - C. from about 5% to about 30% of a hexahydric humectant selected from the group consisting of sorbitol, xylitol, glucose, and mixtures thereof;
 - D. from about 0.1% to about 1% of sodium fluoride; and
 - E. at least about 30% of water,wherein the composition is substantially free of dihydric and trihydric alcohols, and wherein the viscosity of the composition is from about 15 to about 50 BKU.

INTERNATIONAL SEARCH REPORT

International Application No.
PCT/US 00/19580

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A61K7/16 A61K7/18

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the International search (name of data base and, where practical, search terms used)

WPI Data, PAJ, EPO-Internal, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X,P	US 6 045 780 A (BIXLER ET AL.) 4 April 2000 (2000-04-04) the whole document	1-10
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X	US 5 320 830 A (LUKACOVIC ET AL.) 14 June 1994 (1994-06-14) the whole document	1-10
X	EP 0 331 415 A (UNILEVER) 6 September 1989 (1989-09-06) the whole document	1-10
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

14 November 2000

Date of mailing of the international search report

01/12/2000

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INTERNATIONAL SEARCH REPORT

Intern: of Application No
PCT/US 00/19580

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 421 527 A (WASON) 20 December 1983 (1983-12-20) the whole document -----	1-5, 7-9
X	US 5 089 255 A (GAFFAR ET AL.) 18 February 1992 (1992-02-18) the whole document -----	1-3, 5, 7-9

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

Present claims 1-10 relate to a composition defined by reference to the following parameter:

P1: the viscosity of the composition is from about 8 to about 100 BKU

The use of these parameter in the present context is considered to lead to a lack of clarity within the meaning of Article 6 PCT. It is impossible to compare the parameters the applicant has chosen to employ with what is set out in the prior art. The lack of clarity is such as to render a meaningful complete search impossible.

Present claims 1-10 also relate to an extremely large number of possible compositions. Support within the meaning of Article 6 PCT and/or disclosure within the meaning of Article 5 PCT is to be found, however, for only a very small proportion of the compositions claimed. In the present case, the claims so lack support, and the application so lacks disclosure, that a meaningful search over the whole of the claimed scope is impossible. Consequently, the search has been restricted to the examples of the description and in the general concept of the invention.

Claims searched: 1-10 partially

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

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Information on patent family members

Interns Application No
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
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